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(71) Applicant *(for all designated States except US):* AGA AKTIEBOLAG [SE/SE]; S-181 81 Lidingö (SE).

(72) Inventors; and
(75) Inventors/Applicants *(for US only) :* BOKSTRÖM, Monica [SE/SE]; Pargasgatan 12, S-163 27 Spånga (SE). RASIMUS, Raimo [FI/FI]; Suurlohjankatu 33A8, SF-08100 Lohja (FI).

(74) Agent: WIEDEMANN, Bernd; Aga Aktiebolag, S-181 81 Lidingö (SE).

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(54) Title: METHOD FOR WASHING OF ALCALINE PULP

(57) Abstract

Method in the washing of alkaline, cellulosic pulp, for example sulphate pulp, CTMP and CMP pulp, the washing being effected in at least one stage. By an addition of carbon dioxide, there will be realised greatly reduced washing losses of, primarily, inorganic ions, as well as improved washing-out of substances which give rise to chemical oxygen demand (COD).

METHOD FOR WASHING OF ALCALINE PULP

TECHNICAL FIELD

The present invention relates to a method in the washing of alkaline, cellulosic pulp, the washing being effected in one or more consecutive stages.

BACKGROUND ART

In several processes within the cellulose industry, for instance in the manufacture of sulphate pulp and chemi-mechanical pulp, immense volumes of relatively expensive chemicals are employed. In order that these processes be profitable, it is necessary that such treatment chemicals be recycled and regenerated as far as is possible. A further - and now steadily more pressing - reason for the extensive recycling of treatment chemicals is the stringent requirements placed by central and local government authorities on low emissions of chemicals into the environment.

In order to attain washing results which answer to the requirements of such authorities, capital investment costs have had to be offset against running costs. As long as the price of energy was low, it was feasible to increase the volume of washing water, according as the requirements on wash effluent losses became stricter. It was then possible to evaporate the thus obtained large volumes of water. However, now that the price of energy is many times higher, concentrated efforts must be devoted to evolving more efficient washing equipment and more efficient washing processes which, to a considerable degree, free the pulp of its treatment chemicals.

OBJECTS OF THE PRESENT INVENTION

One object of the present invention is to reduce washing losses in the washing of alkaline pulp.

A further object of the present invention is to emasculate alkaline, cellulosic pulp of sodium ions.

A third object of the present invention is to increase the washing-out, from alkaline, cellulosic pulp, of substances which contribute to chemical oxygen demand (COD).

SOLUTION

These objects are attained according to the present invention, in the washing of alkaline, cellulosic pulp, in that, during washing in one or more stages, the pH is lowered in the washing stage or at least one washing stage, the preferred agent added to the washing stage or several washing stages being carbon dioxide.

According to one embodiment of the present invention, the pH is lowered to approximately 9.

According to a further embodiment of the present invention, the pH is lowered by carbon dioxide in one or more washing stages, as a result of which there will be attained an improved washing-out from the pulp of substances which contribute to chemical oxygen demand (COD).

The preferred agent for realising an improved washing of the pulp is carbon dioxide which is added to washing water used in the washing of the pulp and/or to the pulp suspension, prior to the washing stage. Possibly, carbon dioxide may also be added to the washing stage proper.

Carbon dioxide is the particularly preferred agent for attaining improved washing results. Carbon dioxide contains no environmentally hazardous substances such as chlorine or sulphur.

However, in order to lower the pH, other acids may be added, for example sulphuric acid, which, granted, contains sulphur, since the pulp which is washed according to the present invention has been treated - prior to the washing - with sulphate-containing treatment liquid. If used, sulphuric acid is preferably added together with carbon dioxide in order to achieve a more manifest reduction of pH. This said, the pH should not be reduced overly much, since undesirable reactions with residual lignin may then occur.

Above all, the pulp which can be washed with an addition of carbon dioxide or other pH reducing agent is sulphate pulp of soft-wood and deciduous or hardwood, and also chemi-mechanical pulp, CTMP and CMP.

Since the washing operation is normally performed in several stages, the pH reducing agent is added in one or more stages, the agent not being added at least in the first washing stage.

According to a particularly preferred embodiment of the present invention, the pH is reduced in the last stage of, for example three or four washing stages, to approximately 5. In such instance, use may also be made, in addition to carbon dioxide, of a mineral acid, especially sulphuric acid.

BRIEF DESCRIPTION OF THE ACCOMPANYING DRAWINGS

The nature of the present invention and its aspects will be more readily understood from the following brief description of the accompanying Drawings, and discussion relating thereto.

In the accompanying Drawings:

Fig. 1 is a schematic view of a laboratory plant for carrying the present invention into effect; and

Fig. 2 is a schematic view of a slightly modified plant for washing alkaline pulp in four stages.

DESCRIPTION OF PREFERRED EMBODIMENT

Referring to the Drawings, reference numeral 51 in Fig. 1 designates a source for carbon dioxide which, by the intermediary of a conduit 57, is in communication with a rotameter 52, a manometer 53 and a vessel 54. The bottom 58 of the vessel 54 is a sintered body which finely divides the gas entering from the source 51. During the carbon dioxide addition, the contents of the vessel 54 are agitated by means of an agitator 55 driven by a motor 56.

To 25 g of dry pulp, there was added 200 ml of black liquor. The mixture was left to stand for 48 hours. Thereafter, the mixture was diluted with de-ionised water to one litre, i.e. to a 2.5% pulp concentration. The suspension underwent agitation for one hour before the filtrate was removed. The pulp was then subjected to washing in four stages. In each stage, de-ionised water was added to a pulp concentration of 2.5%. Each washing stage lasted for one hour under agitation. All filtrates were kept for analysis. The amount of

sodium in the washing water was determined using atom absorption and COD according to the Dr. Lange method.

In the accounted experimental series, carbon dioxide was added in the second washing stage during 5, 10, 20 and 40 minutes, respectively. The carbon dioxide flow was constant and uniform in all experiments. Because of the low degree of efficiency on the dissolution of carbon dioxide in these experiments, the quantity of CO_2 has not been calculated. In industrial and other plants, equipment well-known to the person skilled in the Art may readily dissolve the carbon dioxide in the washing water and pulp suspension.

It will be apparent from Table 1 that the total amount of washed-out sodium will be higher when CO_2 is added in the 2nd washing stage. The effect becomes manifest already in the stage where the carbon dioxide addition took place, i.e. the 2nd stage. Na^+ is more effectively washed-out as a result of the pH reduction which results from addition of CO_2.

The COD washing-out procedure is more complicated. Total amount of washed-out COD will be higher in an addition of CO_2 in the 2nd washing stage. However, the effect is delayed and the improved washing effect does not become apparent until the 3rd washing stage.

It is not clear why the COD washing-out improves in this way. One possible explanation is of surface chemical nature. The following will then apply:

$(RCOO)_2Ca \rightarrow RCOONa\ (RCOOH)$ resin and fatty acids

$CO_2 + H_2O \rightarrow H_2CO_3$

$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3$

$CaCO_3$ is more sparingly soluble than $(RCOO)_2Ca$

Calcium carbonate is deposited temporarily, calcium being 'inactivated'. Calcium soaps are insoluble in character and do not form lamellar phases to the same extent as sodium soaps. The surface activity in the system increases, which gives an improved washing-out of organic substance.

A modified plant for washing of pulp in sulphate cooking is illustrated in Fig. 2.

According to Fig. 2, the pulp comes from a digester (not shown) through a conduit 1 to a blow tank 3. The conduit 2 leads to a blow condensor. The pulp in the blow tank 3 is diluted with weak (thin) liquor via a conduit 4 from a weak liquor cistern 19. An agitator in the blow tank 3 is designated 5.

The pulp from the blow tank 3 is passed through a conduit 29 to a knotter screen 6. The pulp passes thence to the first washing filter 15 and subsequently to the three following washing filters 16, 17 and 18. The filtrate from the first filter 15 is collected in the weak liquor cistern 19 and the filtrate from the other three filters in the washing liquor cisterns 20, 21 and 22, respectively. The washed pulp departs from the fourth filter 18 at reference numeral 13. When the pulp passes from one filter to the next, the filter cake is comminuted by shredders 8. Filtrate from one filter is used as washing liquid in the preceding filter and dilution liquid in the same filter. Normally, pure water is added to the last filter 18 as washing liquid, entering through a conduit 12.

According to the present invention, carbon dioxide is introduced into the conduits 31 and/or 32 via conduits 23 and 24, respectively. The carbon dioxide dissolves at once in the washing liquid and is led to the filters 17 and 18, respectively through the conduits 31 and 32 and the conduits 10 and 11. The filtrate from the filter 16 passes through a conduit 26 to the cistern 20. A pH sensor device is disposed either in the cistern 20 or in the conduit 26, the sensor controlling, by the intermediary of the control and regulation equipment, the supply of carbon dioxide to the conduit 31. Correspondingly, there is disposed a pH sensor device in the conduit 27 or the cistern 21, this sensor being connected to control apparatus which maintains the pH at the predetermined level by adjusting the CO_2 addition via the conduit 24.

Water is added to the last washing filter 18, this water being admixed with carbon dioxide and/or sulphur dioxide. A pH sensor device may also be disposed in the conduit 28.

It has been found that it is possible to reduce the wash losses, counted as Na_2SO_4, by approx. 1 kg per batched kg of carbon dioxide. A suitable amount of carbon dioxide is approx. 6 kg per tonne of pulp, it being thus possible to reduce the washing loss by approx. 6 kg/tonne of pulp.

By further reducing the pH by an addition of a mineral acid, preferably sulphuric acid, and thus lowering the pH to 6 or less, even lower washing losses will be achieved.

In the manufacture of CTMP and CMP pulp, the cellulosic material is pre-treated with alkaline treatment liquid and is disintegrated in one or more refiners, normally disc refiners. The pulp suspension passes thence to a screen room. The accept therefrom then passes to a washing plant, for example of the type illustrated in Fig. 2.

Using the method according to the present invention, it is possible to improve the washing results of all alkaline, cellulosic pulp, irrespective of whether the pulp is softwood/hardwood pulp or any other type of pulp, for example produced from bagasse.

The present invention should not be considered as restricted to that disclosed above and shown on the Drawings, many modifications being conceivable without departing from the spirit and scope of the appended Claims.

Table 1

CO_2 addition in 2nd washing stage (CO_2 flow constant on addition)

	0 sample	CO_2 5 min	CO_2 10 min	CO_2 20 min	CO_2 40 min
Na (tot) / Na (1)	1.14	1.26	1.23	1.18	3.28
Na (1+2) / Na (1)	1.10	1.21	1.19	1.16	1.22
Na (1+2+3) / Na (1)	1.13	1.25	1.21	1.17	1.24
pH (2)	10.8	7.5	7.4	7.2	5.7
pH (4)	9.4	7.6	7.8	7.6	7.1
COD (tot) / COD (1)	1.20	1.29	1.26	1.32	1.32
COD (1+2) / COD (1)	1.11	1.13	1.13	1.12	1.07
COD (1+2+3) / COD (1)	1.17	1.21	1.19	1.23	1.25

Na (tot) / Na (1) relates to the quotient of total washed-out Na and washed-out Na in the first washing stage. Correspondingly the example below relates to

COD (1+2) / COD (1) COD washed out in first plus second stage through COD washed out in stage 1.

No carbon dioxide was added in stage 1.
pH(2) refers to pH after the second washing stage.

WHAT WE CLAIM AND DESIRE TO SECURE BY LETTERS PATENT IS:

1. A method in the washing of alkaline, cellulosic pulp, in which the washing is effected in one or more consecutive stages, **characterised in that** the pH is lowered in the washing stage, or at least one washing stage.

2. The method as claimed in claim 1, **characterised in that** the pH is lowered by carbon dioxide.

3. The method as claimed in claim 2, **characterised in that** the carbon dioxide is added to the washing water of the stage and/or the pulp suspension immediately prior to the washing stage.

4. The method as claimed in claim 3, in which the washing is effected in at least two stages, **characterised in that** the carbon dioxide is added to all stages except the first stage.

5. The method as claimed in any one or more of claims 1 to 4, **characterised in that** the pH is reduced to at least 9.

6. The method as claimed in claim 5, **characterised in that** the pH is reduced to at least 7.

7. The method as claimed in claim 6, **characterised in that** the pH is reduced to at least 5.

8. The method as claimed in any one or more of claims 1 to 7, **characterised in that** the pulp is sulphate and/or chemi-mechanical (CTMP, CMP) pulp.

9. The method as claimed in claim 1, **characterised in that** the pH is reduced by a mineral acid, preferably sulphuric acid.

10. The method as claimed in claim 4, **characterised in that** a mineral acid, preferably sulphuric acid, is also added to the last washing stage.

Fig. 1

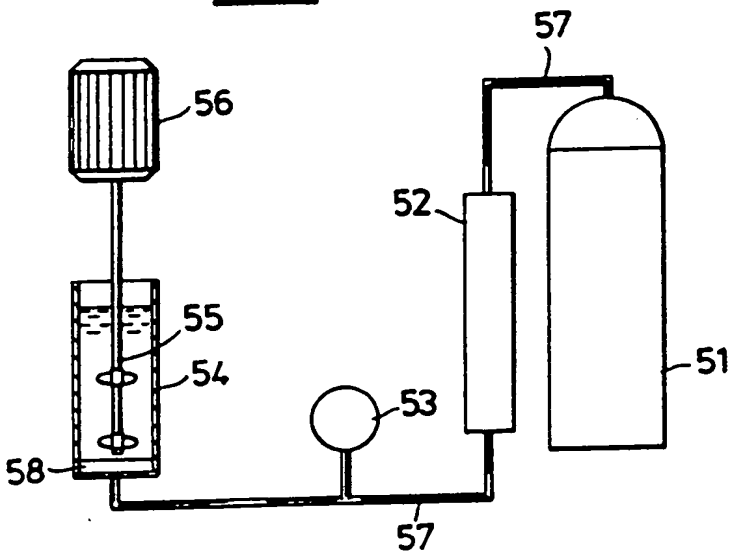
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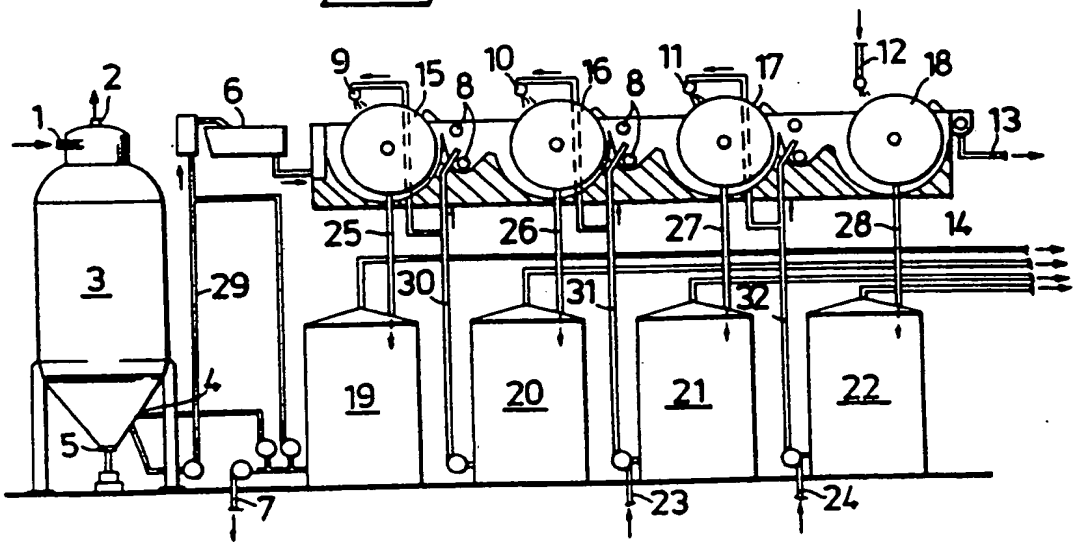
Fig. 2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INTERNATIONAL SEARCH REPORT

International Application No PCT/SE87/00198

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [6]

According to International Patent Classification (IPC) or to both National Classification and IPC

D 21 C 9/02 4

II. FIELDS SEARCHED

Minimum Documentation Searched [7]

Classification System	Classification Symbols
IPC 4	D 21 C 9/02, /04, /06
US Cl	8:156; 162:60

Documentation Searched other than Minimum Documentation to the Extent that such Documents are included in the Fields Searched [8]

SE, NO, DK, FI classes as above

III. DOCUMENTS CONSIDERED TO BE RELEVANT [9]

Category *	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No. [13]
X	SE, B, 420 512 (A. AHLSTRÖM OY) 12 October 1981 & FR, 2283989 AU, 497403 JP, 51049906 CA, 1070058	1, 5, 6, 7, 8, 9
X	Derwent's abstract No 18030 C/10, SU 670 652.	1
X	Derwent's abstract No 91532 C/51, SU 730 913.	1

* Special categories of cited documents: [10]

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
1987-07-10	1987 -07- 23
International Searching Authority	**Signature of Authorized Officer**
Swedish Patent Office	Marianne Bratsberg/ Bo Bergström

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